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(54) **Title:** A PROCESS FOR MAKING RARE EARTH DOPED OPTICAL FIBRE

(57) **Abstract:** The present invention provides an improved process for making rare earth doped preforms and fibres by a combination of MCVD technique and solution doping method, said method comprising developing matched or depressed clad structure inside a silica glass substrate tube followed by deposition of unsintered particulate layer containing GeO_2 and P_2O_5 for formation of the core and solution doping by soaking the porous soot layer into an alcoholic/aqueous solution of RE-salts containing co-dopants like $AlCl_3$ / $Al(NO_3)_3$ in definite proportion, controlling the porosity of the soot, dipping period, strength of the solution and the proportion of the codopants to achieve the desired RE ion concentration in the core and minimise the core clad boundary defects and followed by drying, oxidation, dehydration and sintering of the RE containing porous deposit and collapsing at a high temperature to produce the preform and overladding with silica tubes of suitable dimensions and fibre drawing to produce fibres.

iv. Following immersion, the tube is rinsed with acetone and remounted on lathe.

v. The core layer containing the RE is dehydrated and sintered to produce a clear glassy layer. Dehydration is carried out a temperature of 600°C by using chlorine. The level of OH^- is reduced below 1ppm using Cl_2 / O_2 ratio of 5:2 provided the drying time exceeds 30 min.

vi. Collapsing in the usual manner to produce a solid glass rod called preform.

vii. Fibre drawing is conventional.

Reference may also be made to DiGiovanni D.J., SPIE Vol. 1373 (1990) p-2 "Fabrication of rare-earth-doped optical fibre' wherein the substrate tube with the porous core layer is soaked in an aqueous or alcoholic solution containing a nitrate or chloride of the desired RE ion. The tube is drained, dried and remounted on lathe. The dehydration is carried out by flowing dry chlorine through the tube at about 900°C for an hour. After dehydration, the layer is sintered and the tube is collapsed to be drawn to fibre.

Another reference may be made to Ainslie B.J., Craig S.P., Davey S.T., and Wakefield B., Material Letters, Vol. 6, (1988) p-139, " The fabrication, assessment and optical properties of high- concentration Nd^{3+} and Er^{3+} doped silica based fibres" wherein optical fibres based on Al_2O_3 - P_2O_5. - SiO_2 host glass doped with high concentrations of Nd^{3+} and Er^{3+} have been fabricated by solution method and quantified. Following the deposition of cladding layers P_2O_5 doped silica soot is deposited at lower temperature. The prepared tubes are soaked in an alcoholic solution of 1M $Al(NO_3)_3$ + various concentrations of $ErCl_3$ and $NdCl_3$ for 1 hour. The tubes are subsequently blown dry and collapsed to make preforms in the usual way. Al is said to be a key component in producing high RE concentrations in the core centre without clustering effect. It is further disclosed that Al and RE profile lock together in some way which retards the volatility of RE ion. The dip at the core centre is observed both for P and GeO_2.

Reference may also be made to US Patent No. 5,005,175 (1991) by Desuvire et al., 'Erbium doped fibre amplifier" wherein the fibre for the optical amplifier comprises a single mode fibre doped with erbium in the core having a distribution profile of the RE ion whose radius is less than 1.9 μm while the radius of the mode of the pump signal exceeds 3 μm. The numerical aperture (NA) of the fibres varies from 0.2 to 0.35 and the core is doped with both Al and Ge oxides to increase the efficiency. As the radius of the Er doped core region is equal to or less than the radius of the pump mode of the fibre it is claimed that each atom of erbium in the core cross section is exposed to substantially equal levels of the high intensity portion of the pump mode. The fibre with such design is reported to

phosphorus and as a result the doping concentration of Al ions can be set to a high level (>3 wt%). The dopant concentration and component ratio of Er, Al and P ions are claimed to be extremely accurate and homogeneous in the radial as well as in longitudinal directions.

A few of the drawbacks of the above mentioned processes are as follows:

1. Step like RE distribution profile is obtained in the core resulting to poor overlap between the pump signal and the RE ions which lowers the pump efficiency.
2. Step like RE distribution requires high numerical aperture (NA) of the core or confinement of the RE in the central region (say 50% of the total core area) for increase in pump efficiency which in turn leads to the following disadvantages:

   i) Doping of RE only in selected portion of the core is extremely difficult and affects the repeatability of the process due to the sensitivity of the method to process parameters during various stages of processing such as deposition, solution doping, drying and sintering.

   ii) Increasing the NA of the fibre with simultaneously reducing the core area requires high germania concentration in a small core which enhances the possibility of formation of the dip at the centre due to evaporation during sintering & collapsing.

   iii) For preforms with high NA (>0.20) high germania concentration in the core lowers the viscosity of the glass and makes the process very sensitive to temperature especially during the stages of porous soot layer deposition and sintering.

   iv) Increase in temperature sensitivity during porous soot deposition leads to variation in composition and soot density along the length of the tube.

   v) High germania concentration in the core results to generation of residual stress at the core-clad interface due to difference in viscosity and thermal expansion coefficient. Residual stress produces undesirable increase in background loss of the fibre.

   vi) Residual stress is believed to introduce polarisation mode dispersion (PMD) which results in serious capacity impairments including pulse broadening. Since the magnitude of PMD at a given wavelength is not stable passive compensation becomes impossible.

3. Dehydration and sintering of the RE chloride containing soot layer is critical because it alters the composition by vaporisation and also diffusion of the dopant salt as well as GeO_2 present in the core.

inventive step lies in transformation of the RE salts to oxides by gradually heating the tube to a higher temperature maintaining an oxidising atmosphere inside, thereby minimising the possibility of evaporation of RE during subsequent processing as the oxide has a very high melting temperature compared to halide/nitrate salts. This step also helps to remove the solvent trapped within the porous layer. The inventive step also includes increasing the temperature of the RE containing porous layer gradually in steps of 50 to 200°C up to the sintering temperature and above for sintering and further fixing of the RE ions in their desired sites. The steps will depend on the host glass composition and Er/Al concentration of the core layer. The incorporation efficiency of the RE from the solution to the core layer thus increases appreciably making the process more efficient and economic. The RE distribution along the transverse direction in the core will depend on the density of the porous soot layer, dipping period and the processing conditions during oxidation, sintering and collapsing.

The sintering of the porous core layer in GeO_2 rich atmosphere along with the addition of oxygen and helium is another inventive step of the process which reduces the quantity of $GeCl_4$ required to achieve the desired NA and adds to the economy of the process. At temperatures between 200° to 1400° C during the sintering step pure $GeCl_4$ is supplied with the input oxygen, the quantity of which depends on the NA desired in the fibre. The sintering is continued by gradually raising the temperature till a clear glassy layer is formed.

Detailed Description of the Invention

Accordingly the present invention provides an improved process for making rare earth doped optical fibre which comprises (a) providing deposition of P_2O_5 and F doped synthetic cladding within a silica glass substrate tube to obtain matched or depressed clad type structure, (b) forming a core by depositing unsintered particulate layer at a tube surface temperature in the range of 1200-1400°C, (c) maintaining P_2O_5 and GeO_2 concentrations from 0.5 to 5.0 mol% and 3.0 to 25.0 mol% in the said particulate layer respectively to obtain a tube containing F-doped cladding and porous soot layer, (d) immersing the tube containing the porous soot layer into a solution containing RE salt in the concentration range of 0.002M to 0.25 M with or without aluminium salt of the concentration range 0.05 M to 1.25 M for a period of 1 to 2 hours, (e) draining the solution out at a rate in the range of 10-50 cc/min, (f) drying the porous layer by flowing dry nitrogen or any other inert gas through the tube, (g) heating the tube gradually in the presence of oxygen at a temperature in the range of 600-1100°C, (h) dehydrating the core

In another embodiment of the present invention $GeCl_4$ supplied during soot deposition is 10 to 30% less than that required for achieving the desired NA numerical aperture.

In another embodiment of the invention, the pump beam has a radius of distribution equal to or greater than the radius of distribution of Er ions in the core, which enhances the chance of all the active ions getting exposed to the pump light.

In another embodiment of the invention, relatively high gain is achieved in the fibres for NA (Numerical aperture) value close to 0.20.

In yet another embodiment of the present invention RE salt used is selected from chloride, nitrate or any other salt soluble in solvent used in the process.

In still another embodiment of the present invention aluminium salt used is selected from chloride, nitrate or any other salt soluble in solvent used in the process.

In yet another embodiment of the present invention solution for aluminium and RE salts is prepared using solvent selected from alcohol and water.

In still another embodiment of the present invention the temperature of the core layer is increased in steps of 50 to 200°C during oxidation and sintering depending on the composition and Al/RE concentration of the core layer.

In yet another embodiment of the present invention the mixture of O_2 and He is in the range of 3:1 to 9:1.

In still another embodiment of the present invention source of chlorine is selected from CCl_4 where Helium is used as carrier gas.

In yet another embodiment of the present invention the proportion of Cl_2: O_2 varies from 1.5: 1 to 3.5: 1 while the dehydration period lies between 1 to 2 hours.

In yet another embodiment of the present invention the porous core is sintered in presence of germania by supplying $GeCl_4$ with the input oxygen at a temperature of 1200°C to 1400°C during sintering to facilitate germania incorporation and obtain appropriate numerical aperture.

In yet another embodiment, the process provides variation in the numerical aperture of the fibre from 0.10 to 0.30 maintaining RE concentration in the core between 50 to 6000 ppm along with variation in RE distribution profile along the transverse direction in the doped region to produce fibres suitable for application in any devices.

In yet another embodiment, the devices are amplifiers, fibre lasers and sensors for different purposes where optical fibre is used.

Another embodiment of the invention is a method of controlling the Gaussian RE distribution profile along the radial direction in a core used in the process of making rare

j) the core layer from the drying temperature between 800 to 1200°C mentioned above.

k) At temperatures between 1200° to 1400°C during sintering pure $GeCl_4$ is supplied with the input oxygen to carry out the sintering of the porous layer in germania rich atmosphere which facilitates germania incorporation. The flow rate of $GeCl_4$ and the no. of pass depend on the NA desired in the fibre. The supply of $GeCl_4$ is then stopped and the sintering is continued by gradually raising the temperature till a clear glassy layer is formed.

l) The collapsing is carried out at a high temperature (>2000°C) in 3 to 4 passes of the burner to produce a solid glass rod called preform.

m) The preform is overcladded with silica tubes of suitable dimensions to achieve the appropriate core - clad dimensions in the ultimate preform/fibre.

n) Fibres are drawn from the preform.

Brief description of the accompanying drawings

Figure 1 & 2 represents Er fluorescence distribution across the fibre core

The invention is further explained with the help of following examples, which should not be construed to limit the scope of the invention:

EXAMPLE 1

- Deposition of F-doped cladding within a silica tube by MCVD process at a temperature of 1855°C.
- Unsintered core deposition at a temperature of 1290°C. The carrier gas flows through the reagent liquids were adjusted to obtain a composition of SiO_2= 90.2 mol%, P_2O_5= 1.3 mol% and GeO_2= 8.5 mol% in the deposited soot layer.
- Dipping the tube with the deposited layer in a solution containing 0.025 (M) $ErCl_3$ and 0.15 (M) $Al(NO_3)_3\ 9H_2O$ for 1 hour and draining out the solution slowly.
- Drying by maintaining nitrogen gas flow through the tube for 10 min.
- Oxidation at temperatures of 725°C, 825°Cand 950°C with 2 passes of the burner at each temperature maintaining a constant He/O_2 ratio of 1:5.
- Dehydration was carried out at a temperature of 1010°C with a Cl_2: O_2 ratio of 2.5: 1 for a period of 1 hour 15 mins.
- The temperature was increased in 4 steps up to1400°C. $GeCl_4$ was added from this stage with input oxygen with 3 passes between 1200° 1400°C. The tube was further heated to increase the temperature stepwise to 1650°C for complete sintering of the Er

- The Er^{3+} ion concentration in the fibre was 460 ppm with peak at the core centre and similar distribution as shown in accompanying drawings as figure 1.
- The fibre recorded a gain up to 37 dB as measured from C-DOT, 39 Main Pusa Road, New Delhi – 110 005 using their measurement set-up.

EXAMPLE 3

- Deposition of F-doped cladding within a silica tube by MCVD process at a temperature of 1870°C.
- Unsintered core deposition at a temperature of 1250°C. The carrier gas flows through the reagent liquids were adjusted to obtain a composition of SiO_2= 89.1 mol%, P_2O_5= 2.3 mol% and GeO_2= 8.6 mol% in the deposited soot layer.
- Dipping the tube with the deposited layer in a aqueous solution containing 0.07 (M) $ErCl_3$ and 0.25 (M) $Al(NO_3)_3$ $9H_2O$ for 1 hour and draining out the solution slowly.
- Drying by maintaining nitrogen gas flow through the tube for 10 min.
- Oxidation at temperatures of 730^0, 820° and 925°C with 2 passes of the burner at each temperature maintaining at constant He/O_2 ratio of 1:6.
- Dehydration was carried out at a temperature of 925°C with a Cl_2: O_2 ratio is 2.3 : 1 for a period of 1.5 hour.
- The temperature was increased in 4 steps up to1400°C. $GeCl_4$ was added with the input oxygen with 2 passes at 1200^0C and one pass each at 1300^0C and 1400^0C. The tube was further heated to increase the temperature stepwise to 1725°C for complete sintering of the Er & Al containing porous soot layer. During sintering O_2 and He flow was in the ratio of 4:1.
- The collapsing was done in 3 steps in the usual manner.
- Overcladding was done to reduce the core:clad ratio to 6.5:125. The NA measured in the fibre was 0.22±.01 .
- The Er^{3+} ion concentration in the fibre was 3020 ppm with peak concentration at the core centre and Er distribution in the core as shown in accompanying drawing as figure -2 measured from the fibre section by fluorescence spectroscopy by Photonics Resource Facility, 60 St. George Street, Suite No. 331, Toronto, Ontario, Canada M5S-1A7.

The main advantages of the present invention are:

1. The developed fibres have a RE distribution in the doped region similar to the Gaussian pump beam intensity distribution in the fibre so that the overlapping between

12. The concentration of RE in the core is varied between 50 to 6000 ppm along with variation in RE distribution profile in the doped region and NA between 0.10 to 0.30 to produce fibres suitable for application as amplifiers, microlasers and sensors for different purposes.

6. A process as claimed in claim 1 wherein, the mixture of O_2 and He is in the range of 3:1 to 9:1.
7. A process as claimed in claim 1 wherein, the source of chlorine is CCl_4 where He is used as carrier gas.
8. A process as claimed in claim 1 wherein, the proportion of Cl_2: O_2 is ranging from 1.5: 1 to 3.5: 1 while the dehydration period lies between 1 to 2 hours.
9. A process as claimed in claim 1 wherein, during sintering of porous core layer $GeCl_4$ is supplied with the input oxygen maintaining a temperature of 1200°C to 1400°C.
10. A process as claimed in claim 1 wherein, sintering in germania rich atmosphere facilitates higher germania incorporation and reduces the quantity of germanium halide necessary during deposition.
11. A process as claimed in claim 1 wherein, the oxidation step before drying and sintering of the particulate layer reduces the possibility of change in composition due to evaporation of RE salts during subsequent processing.
12. A process as claimed in claim 1 wherein, the increase in temperature in steps of 50 to 200°C during oxidation and sintering stages prevents diffusion of RE and the codopants from the doped region resulting to minimum change in composition.
13. A process as claimed in claim 1 where the incorporation efficiency of RE in the doped region is increased.
14. An improved process for producing Er doped fibre in particular characterised by Er ion distribution in the core similar to the Gaussian pump beam intensity distribution wherein, the said process comprising steps of
    (a) providing deposition of P_2O_5 and F doped synthetic cladding within a silica glass substrate tube to obtain matched or depressed clad type structure,
    (b) forming a core by depositing unsintered particulate layer at a tube surface temperature in the range of 1200-1350°C,
    (c) maintaining P_2O_5 and GeO_2 concentrations from 0.5 to 3.5 mol% and 3.0 to 20.0 mol% in the said particulate layer respectively to obtain a tube containing F-doped cladding and porous soot layer,
    (d) immersing the tube containing the porous soot layer into a solution containing Er salt in the concentration range of 0.004 M to 0.20 M with or without aluminium salt in the concentration range 0.05 M to 1.0 M for a period of 1 to2 hours,
    (e) draining the solution out at a rate in the range of 10-30 cc/min,

24. A process as claimed in 14 wherein, the pump beam has a radius of distribution equal to or greater than the radius of distribution of Er ions in the core, which enhances the chance of all the active ions getting exposed to the pump light.
25. A process as claimed in 24 wherein, relatively high gain is achieved in the fibres for NA value close to 0.20.
26. A process as claimed in 14 wherein, wide variation in composition between the core and cladding glass is avoided due to relatively low NA of the fibre eliminating problems like residual stress and PMD, which may substantially degrade the performance of the fibre.
27. A process as claimed in 14 wherein, the compositions of the core and cladding glass are suitable to achieve NA of 0.20 and Er^{3+} ion concentration in the range of 100 to 1500 ppm without clustering in order to provide EDF suitable for pumping at 980 nm for amplification of the input signal with gain in the range 10 to 37 dB for optical amplifier application.
28. A process as claimed in 14 wherein, the developed fibres have NA and mode field diameter not widely different from signal transmitting fibre for ease of splice and this minimises the optical loss of the signal travelling through the fibres.
29. A process as claimed in 14 wherein, sintering in germania rich atmosphere reduces the quantity of germanium halide necessary during deposition to achieve the desired NA.
30. A process as claimed in 14 wherein, the oxidation step before drying and sintering of the particulate layer reduces the possibility of change in composition due to evaporation of Er salts during subsequent processing.
31. A process as claimed in 14 wherein, the increase in temperature in steps of 50 to 200°C during oxidation and sintering stages prevents diffusion of RE and the codopants minimising the possibility of change in composition.
32. A process as claimed in 14 wherein, the incorporation efficiency of RE in the doped region is increased which in turn increases the economy and repeatability of the process.
33. A process as claimed in 14 wherein, the numerical aperture of the fibre is varied from 0.10 to 0.30 maintaining Er concentration in the core between 50 to 6000 ppm along with variation in Er distribution profile in the doped region to produce fibres suitable for application as amplifiers, fibre lasers and sensors for different purposes.
34. A method of controlling the Gaussian RE distribution profile along the radial

41. A process as claimed in claim 34 the proportion of Cl_2: O_2 is varying from 1.5: 1 to 3.5: 1 while the dehydration period lies between 1 to 2 hours.
42. A process as claimed in claim 34 wherein, during sintering of porous core layer $GeCl_4$ is supplied with the input oxygen maintaining at a temperature ranging from 1200°C to 1400°C.
43. A process as claimed in claim 34 wherein, the oxidation step before drying and sintering of the particulate layer reduces the possibility of change in composition due to evaporation of Er salts during subsequent processing.
44. A process as claimed in claim 34 wherein, the stepwise increase in temperature during oxidation and sintering stages prevents diffusion of RE and the codopants which in turn prevents change in composition.
45. A process as claimed in 34 wherein, the numerical aperture of the fibre is varied from 0.10 to 0.30 maintaining RE concentration in the core between 50 to 6000 ppm along with variation in the RE distribution profile in the doped region to produce fibres suitable for any devices.
46. A process as claimed in claim 34, wherein the devices are amplifiers, fibre lasers, and sensors for different purposes and other devices where optical fibre is used.

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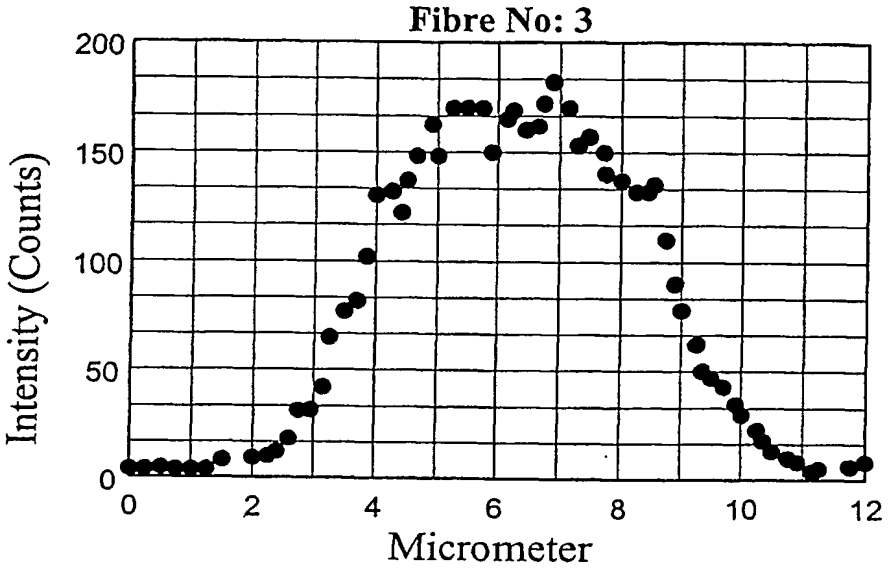

Fig. 2: Er fluorescence distribution across the fibre core.

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
X	EP 1 043 281 A (SPECTRAN CORP) 11 October 2000 (2000-10-11)	1-9,11, 13-21, 23-28, 30,32,33
A	the whole document	10,12, 22,29, 31,34

X	TOWNSEND J E ET AL: "solution-doping technique for fabrication of rare-earth-doped optical fibres" ELECTRONICS LETTERS, IEE STEVENAGE, GB, vol. 23, no. 7, 26 March 1987 (1987-03-26), pages 329-331, XP002109622 ISSN: 0013-5194 cited in the application	1-9,11, 13-21, 23-28, 30,32,33
A	the whole document	10,12, 22,29, 31,34

X	CARTER A L G ET AL: "FLASH-CONDENSATION TECHNIQUE FOR THE FABRICATION OF HIGH-PHOSPHORUS-CONTENT RARE-EARTH-DOPED FIBRES" ELECTRONICS LETTERS, IEE STEVENAGE, GB, vol. 28, no. 21, 8 October 1992 (1992-10-08), pages 2009-2011, XP000320367 ISSN: 0013-5194	1,14
A	the whole document	34

A	DIGIOVANNI D J ET AL: "THE EFFECT OF SINTERING ON DOPANT INCORPORATION IN MODIFIED CHEMICAL VAPOR DEPOSITION" JOURNAL OF LIGHTWAVE TECHNOLOGY, IEEE. NEW YORK, US, vol. 7, no. 12, 1 December 1989 (1989-12-01), pages 1967-1972, XP000103655 ISSN: 0733-8724 the whole document	1,12,14, 31,34
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(57) Abstract: The present invention provides an improved process for making rare earth doped preforms and fibres by a combination of MCVD technique and solution doping method, said method comprising developing matched or depressed clad structure inside a silica glass substrate tube followed by deposition of unsintered particulate layer containing GeO_2 and P_2O_5 for formation of the core and solution doping by soaking the porous soot layer into an alcoholic/aqueous solution of RE-salts containing co-dopants like $AlCl_3$ / $Al(NO_3)_3$ in definite proportion, controlling the porosity of the soot, dipping period, strength of the solution and the proportion of the codopants to achieve the desired RE ion concentration in the core and minimise the core clad boundary defects and followed by drying, oxidation, dehydration and sintering of the RE containing porous deposit and collapsing at a high temperature to produce the preform and overladding with silica tubes of suitable dimensions and fibre drawing to produce fibres.